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2-(4'-Alkoxyphenyl)-5-alkylpyridines.

Novel liquid crystal compounds exhibiting an SC phase which, when added to an optically active liquid crystal, give a chiral smectic liquid crystal material of use in liquid crystal display elements utilizing the ferroelectricity of the material have the formula:

$$R^1 - O_N - O_N^2$$

wherein R1 represents a C7C12 alkyl group and R2 represents C5-C12 alkyl group.

## Description

## 2-(4'-Alkoxyphenyl)-5-alkylpyridines

#### BACKGROUND OF THE INVENTION

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#### I. Field of the Invention

This invention relates to a novel liquid crystal compound and a chiral smectic C liquid crystal composition containing the above-mentioned liquid crystal compound as a constituent thereof and useful for liquid crystal display elements.

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#### 2. Description of the Relates Art

At present, as to liquid crystal display elements, TN (Twisted Nematic) type display mode has been most broadly employed, but as far as the response speed is concerned, such TN type display elements are inferior to emissive type display elements (such as those of electroluminescence, plasma display, etc.). Although various improvements in this respect have been attempted, it appears that possibility of the improvement to a large extent has not been left behind so much. Thus, various liquid crystal display devices based on a different principle from that of TN type display elements have been attempted. As one of such devices, there is a display mode utilizing a ferroelectric liquid crystal (N.A. Clark et al: Applied Phys. lett., <u>36</u>, 899 (1980)). This mode utilizes the chiral smectic C phase (hereinafter abbreviated to SC\* phase) or the chiral smectic H phase (hereinafter abbreviated to SH\* phase) of the ferroelectric liquid crystal, and those having these phases in the vicinity of room temperature are preferred.

These chiral smectic liquid crystal materials may be obtained by blending a plurality of single compounds each exhibiting a chiral smectic phase by itself, but it is known that the materials may be also obtained by adding an optically active liquid crystal compound, preferably a chiral smectic liquid crystal compound to an achiral smectic liquid crystal exhibiting smectic C phase (SC phase), smectic H phase (SH phase), etc.

Various kinds of compounds exhibiting SC phase have already been known, but as to whether or not chiral smectic liquid crystal materials obtained by adding thereto an optically active liquid crystal exhibit superior performances in liquid crystal display utilizing ferroelectricity, no ultimate evaluation thereof has been yet obtained. This is due to the fact that liquid crystal display utilizing ferroelectricity has not yet been technically completed. Thus, in the present situation, it is necessary to test various novel SC materials.

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## SUMMAY OF THE INVENTION

The main object of the present invention is to provide a novel liquid crystal compound exhibiting SC phase and suitable to the above-mentioned use application.

The present invention resides in

a liquid crystal compound expressed by the formula

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$$R^1 - O_N - OR^2$$
 (I)

wherein R¹ represents an alkyl group of 7 to l2 carbon atoms and R² represents an alkyl group of 5 to l2 carbon atoms, and a chiral smectic liquid crystal composition containing, at least one member of said liquid crystal compounds and at least one kind of optically active liquid crystal compound.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

The phase transition points of the compounds of the formula (I) are shown in Table I.

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Table 1

Sample No.	In form R <sup>1</sup> C <sub>7</sub> H <sub>15</sub>	In formula (I) Rl R <sup>2</sup> C <sub>7</sub> H <sub>15</sub> C <sub>5</sub> H <sub>11</sub>	C SH SG SF SB SC SA N I
2	C7H15	C <sub>6</sub> H <sub>13</sub>	· 34.0 (· 31.2) · 44.4 · 53 74.4 75.2 ·
С	C7H15	C7H15	. 24.0 . 31.5 . 40.3 . 53 76.6
4	C <sub>7</sub> II <sub>15</sub>	C81117	. 45.0 45.4 . 56.5 80.4
5	C7H15	C <sub>9</sub> H <sub>19</sub>	. 42.5 58.5 80.7
9	C7H15	C10H21	. 39.5 62.0 82.1
L	C7H15	C11H23	. 48.8 63.7 81.4
æ	С7H15	C12H25	. 49.0 67.0 82.1
6	C8H17	$c_{5}^{H_{11}}$	. 37.4
10	C8H17	C6H13	. 13.8 .*(Sx) . 19.0 . 54.0 76.2
11	C <sub>8</sub> H <sub>1</sub> 7	C <sub>7</sub> H <sub>15</sub>	. 27.3

Table 1 (continued)

	н		•	•	•	•		•				•
Again the state of	Z						74.5					
(o°)	SA	3	4		7	1	•		10	-	6	3
points (	SC	. 81.	- 81.4	. 82.1	. 82.2	- 82.4	0 . 72.4	4 . 80.5	.0 .81.5	.0 85.	.6 .84.9	2 . 86.
	SB						. 65.0	. 64.	. 64	. 99 .	. 69	. 69 .
se transition	SF	. 60.2	- 61.5	. 65.5	0.89.	. 70.2						
Phase	SG											
	SH											
	ט	. 34.6	. 37.5	. 40.8	. 47.3	. 46.0	. 42.5	. 36.0	. 33.0	. 31.6	. 37.4	. 38.6
In formula (I)	R2	C8H17	C <sub>9</sub> H <sub>19</sub>	C10H21	$c_{11}^{II}$ 23	C12H25	C5H11	C <sub>6</sub> H <sub>13</sub>	C7H15	C <sub>8</sub> H <sub>1</sub> 7	C <sub>9</sub> H <sub>19</sub>	$c_{10}^{H_{21}}$
In form	$_{ m R}^{ m l}$	C <sub>8</sub> H <sub>17</sub>	C <sub>8</sub> H <sub>17</sub>	C8H17	C <sub>8</sub> 11 <sub>17</sub>	C <sub>8</sub> H <sub>1</sub> 7	C <sub>9</sub> H <sub>19</sub>	C <sub>9</sub> H <sub>19</sub>	C9H19	C9H19	C9H19	$C_9H_{19}$
Sample	ON	12	13	1.4	1.5	16	1.7	1.8	19	20	21	22

Table 1 (continued)

Phase transition points (°C)	SH SG SF SB SC SA N I	40.7	43.5 77.8 · 85.2	44.4	30.00	41.0 67.8 · 80.8	34.5	40.7 72.4 84.0	41.3 75.8 85.4	47.6	48.0 79.9 86.1	
	ບ	• 40	• 43	• 44	• 30	. 41	. 34	. 40	. 41	- 47	. 48	+
ula (I)	R <sup>2</sup> .	$c_{11}^{H}$ 23	C12H25	$c_{5}$ H11	C <sub>6</sub> 113	C7H15	C <sub>8</sub> H <sub>1</sub> 7	C9H19	C10H21	C11H23	C12H25	
In formula (I)	$^{\mathrm{R}1}$	$^{\mathrm{C_9H_{19}}}$	$^{\mathrm{C_9H_{19}}}$	$c_{10}{}^{\rm H}_{21}$	C10 <sup>II</sup> 21	C10H21	C10H21	C10H21	C10H21	C10H21	C <sub>10</sub> H <sub>21</sub>	
Sample	No.	23	24	25	26	27	28	29	30	31	32	

Sx means an unknown smectic phase

The compounds of the formula (I) of the present invention exhibit smectic C (SC) phase, and by blending the compound of the formula (I) mentioned later with other optically active liquid crystal compounds, particularly,

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chiral smectic liquid crystal compounds, it is possible to obtain a chiral smectic C liquid crystal composition exhibiting SC\* phase within a broader temperature range. In this case, it is generally preferred to use a plurality of the compounds of the formula (I), and similar compounds exhibiting smectic C phase may be contained in addition to the compound of the formula (I).

Further, as the optically active liquid crystal compounds to be blended with those of the present invention, those exhibiting chiral smectic C phase are preferred, but even in the case of compounds exhibiting no chiral smectic C phase, there may be compounds affording a liquid crystal composition exhibiting chiral smectic C phase by blending such compounds with those of the present invention.

Here, homologues having a similar structure to that of the compound of the formula (I) of the present invention, that is, compounds the respective carbon numbers of  $R_1$  and  $R_2$  of which are outside the ranges of those of the compounds of the formula (I) of the present invention have been reported in a few literatures. Thus, the relationship between these compounds and those of the present invention will be described below. Pavulchenko et al (L. Bata Ed.; Advances in Liquid Crystal Research and Application; Pergamon Press, 1980, page 1007) prepared compounds of the formula (I) wherein  $R^1 = C_4H_9$  and  $R^2 = CH_3$  or  $C_4H_9$  and those wherein  $R^1 = C_6H_{13}$  and  $R_2 = CH_3$ ,  $C_4H_9$ ,  $C_5H_{11}$ ,  $C_6H_{13}$  or  $C_7H_{15}$  (seven kinds of compounds in total), and reported that among these compounds, those except for a compound of  $R^1 = C_6H_{13}$  and  $R^2 = C_5H_{11}$  exhibited at most one kind of smectic modification (however, what a smectic phase the compound exhibits has not been decided), and a compound of  $R^1 = C_6H_{13}$  and  $R^2 = C_5H_{11}$  exhibited the following three kinds of smectic modifications ( $S_1$  to  $S_3$ ):

and presumed that  $S_1$  might perhaps be SA and  $S_2$  might perhaps be SC. However, according to the research of the present inventors, this substance exhibits the following phase transitions:

Further, Japanese patent application laid-open No. Sho 6l-24570/l986 (filed by Suwa Seiko Company and laid-open on February 3, l986) discloses six compounds (a compound of  $R^1 = C_2H_5$  and  $R^2 = C_6H_{13}$ ; that of  $R^1 = C_5H_{11}$  and  $R^2 = C_2H_5$ ; that of  $R^1 = C_5H_{11}$  and  $R^2 = C_6H_{13}$ ; that of  $R^1 = C_5H_{11}$  and  $R^2 = C_6H_{13}$ ; and that of  $R^1 = C_6H_{13}$  and  $R^2 = C_4H_9$ , and indicates that these compounds are nematic liquid crystals or nonliquid crystalline compounds.

However, from such prior art it is not presumed that the compound of the formula (I) of the present invention exhibits SC phase.

Compounds of the formula (I) may be suitably prepared through the following passageways:

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CLCH=CHC OCH<sub>3</sub>

(I)

$$(C_2H_5)_3N$$
 $(C_2H_5)_3N$ 
 $(C_2H_5)_3N$ 

Namely, a known substance p-methoxyphenyl  $\beta$ -chlorovinyl ketone (II) is reacted with an enamine in the presence of triethylamine in a solvent and further reacted with perchloric acid to obtain a pyrylium perchlorate

(III). The compound (III) is reacted with ammonium acetate in a solvent to obtain a compound (IV), which is then reacted with hydrobromic acid in a solvent (preferably, acetic acid) to obtain a compound (V), which is then heated together with an alkylbromide (or an alkyl tosylate) and potassium hydroxide to obtain the objective compounds of the formula (I).

The liquid crystal compound and liquid crystal composition of the present invention will be described in more detail by way of Examples.

#### Example i

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Preparation of 2-[p-(octyloxy)phenyl]-5-heptylpyridine (a compound of the formula (I) wherein  $R^1 = C_7H_{15}$  and  $R^2 = C_8H_{17}$ ; sample No. 4)

(i) Preparation of 5-heptyl-2-(p-methoxyphenylpyrylium)perchlorate

N-heptenylpiperidine (4i.8 g, 0.20 mol) and triethylamine (20 g, 0.20 mol) were dissolved in ethyl ether (l00 m $\ell$ ) with stirring, followed by dropwise adding to the solution, a solution of p-methoxyphenyl  $\beta$ -chlorovinyl ketone (II) (39 g, 0.20 mol) and ethyl ether (200 m $\ell$ ) while keeping the temperature of the system at 35°C or lower, agitating the mixture at room temperature for 8 hours, adding water (l00 m $\ell$ ) and toluene (50 m $\ell$ ), washing the resulting organic layer with water, distilling off the solvent under reduced pressure, adding to the residue, 70% perchloric acid (l00 m $\ell$ ), then adding water (l00 m $\ell$ ), keeping the mixture under reflux for minutes, cooling the resulting material to obtain crystals, washing the crystals with ethyl ether and drying them to obtain 5-heptyl-2-(p-methoxyphenylpyrylium) perchlorate (48.0 g).

(ii) Preparation of 5-heptyl-2-(p-methoxyphenyl)pyridine

5-Heptyl-2-(p-methoxyphenylpyrylium)perchlorate (48.0 g, 0.125 mol), ammonium acetate (96 g, 1.25 mol) and acetic acid (500 m $\ell$ ) were agitated under reflux for 4 hours, followed by pouring the resulting reaction fluid in water, dissolving the resulting crystals in toluene, washing them with water, distilling off the solvent under reduced pressure and recrystallizing the residue from methanol to obtain 5-heptyl-2-(p-methoxyphenyl)pyridine (a compound of the formula (IV) wherein R<sup>1</sup> = C<sub>7</sub>H<sub>15</sub>) (20 g), having a m.p. of 54.4 - 56.5° C.

In addition, compounds of the formula (IV) wherein  $R^1$  represents  $C_8H_{17}$ ,  $C_9H_{19}$  or  $C_{10}H_{21}$  had the following melting points:

5-octyl-2-(p-methoxyphenyl)pyridine m.p. 60.7-62.2° C 5-nonyl-2-(p-methoxyphenyl)pyridine m.p. 55.0-57.4° C 5-decyl-2-(p-methoxyphenyl)pyridine m.p. 6l.l-62.9° C.

35 (iii) Preparation of 5-heptyl-2-(p-hydroxyphenyl)pyridine

To 5-heptyl-2-(p-methoxyphenyl)pyridine (20 g, 0.07l mol) obtained in the above step (ii) (20 g, 0.07l mol) were added hydrobromic acid (47%) (I30 m $\ell$ ) and acetic acid (300 m $\ell$ ), followed by keeping the mixture under reflux for 30 hours, cooling the resulting material, pouring it in water, filtering off the resulting crystals, dissolving the crystals in 2N-NaOH aqueous solution, further adding acetic acid to make the solution acidic, filtering off the deposited crystals and recrystallizing them from a mixture of methanol and water to obtain 5-heptyl-2-(p-hydroxyphenyl)pyridine (a compound of the formula (V) wherein R<sup>1</sup> = C<sub>7</sub>H<sub>15</sub>) (I3 g) having a m.p. of I05.2 - I05.9°C.

In addition, compounds of the formula (V) wherein  $R^1 = C_8H_{17}$ ,  $C_9H_{19}$  or  $C_{10}H_{21}$  had the following melting points:

5-octyl-2-(p-hydroxyphenyl)pyridine m.p. 93.2-95.0°C 5-nonyl-2-(p-hydroxyphenyl)pyridine m.p. 85.0-87.5°C 5-decyl-2-(p-hydroxyphenyl)pyridine m.p. 90.4-9l.8°C

(iv) Preparation of 2-[p-(octyloxy)phenyl]-5-heptylpyridine

5-Heptyl-2-(p-hydroxyphenyl)pyridine (l0 g, 0.037 mol) obtained in the step (iii) together with ethanol (l00 mℓ), potassium hydroxide (3.0 g, 0.045 mol) and octylbromide (9g, 0.048 mol) were heated under reflux with stirring for 4 hours, followed by cooling the resulting material, adding water and toluene, washing the resulting organic layer with 2N-NaOH aqueous solution, then washing it with water, distilling off the solvent under reduced pressure and recrystallizing the residue from ethanol to obtain the objective final product 2-[p-(octyloxy)phenyl]-5-heptylpyridine (6.5 g).

This product had a C-SG point of 45.0°C, a SG-SF point of 45.4°C, a SF-SC point of 56.5°C and a SC-I point of 80.4°C.

The same procedure as above was carried out except that N-heptenylpiperidine used in the step (i) was replaced by other N-alkenylpiperidines and octylbromide used in the step (iv) was replaced by other alkylbromides or alkyl tosylates, to obtain all other compounds of the formula (i).

Example 2 (Composition example I)

Five kinds of compounds of the present invention (sample Nos. 4, 7, I2 and 32) and a compound of the formula (I) having wherein  $R^1 = C_7H_{15}$  and  $R^2 = C_4H_9$ , which compound has a similar chemical structure, but does not fall in the scope of the present invention and has the following phase transition points:

the tilt angle was 24°C.

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were blended in equal quantities to prepare a liquid crystal composition A.

This liquid crystal composition A had a m.p. of I2°C; formed SB phase on the higher temperature side relative to the phase; formed SC phase at 52°C; formed SA phase at 75°C; and formed an isotropic liquid at 78°C.

To 60% by weight of this liquid crystal composition A were added and blended the following two kinds of chiral smectic liquid crystal compounds each in 20% by weight to prepare a chiral smectic liquid crystal composition B:

$$C_6H_{13}$$
 $C_6H_{13}$ 
 $C_6H_{13}$ 

The resulting chiral smectic liquid crystal composition B had a m.p. of -l0°C; exhibited SC\* phase in the temperature range higher than this temperature; formed SA phase at 54°C; and formed an isotropic liquid at 68°C. Its supercooled state was observed down to -l5°C; it had SC\* phase down to this temperature; and no other smectic phase was observed. In addition, the spontaneous polarization value was l0 nC/cm² at 25°C and

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A blend of the above compounds C and D in equal quantities had the following phase transition points:

The above-mentioned chiral smectic liquid crystal composition B was filled in a cell of 2  $\mu$ m thick provided with transparent electrodes having PVA (polyvinyl alcohol) as an aligning agent applied thereonto and subjected to parallel aligning treatment by rubbing the surface, and the resulting liquid crystal element was provided between two sheets of crossed polarizers and an electric field was impressed so that change in the intensity of transmitted light was observed by impression of I5 V.

Response time was sought from the change in the intensity of transmitted light at that time of give about 100 µsec. at 25°C.

As described above, it is seen that by blending the compound of the formula (I) of the present invention with an optically active liquid crystal compound, it is possible to obtain a ferroelectric chiral smectic C liquid crystal composition having a broad range of SC\* phase and superior response properties.

## Example 3 (Composition example 2)

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Seven kinds of the compound of the present invention (sample Nos. 2, 4, II, I3, I8, 22 and 24) were blended together in equal quantities to prepare a liquid crystal composition.

This liquid crystal composition was smectic C composition and its phase transition points were as follows:

When this smectic C composition is blended with an optically active liquid crystal compound, it is possible to obtain superior chiral liquid crystal compositions as in the case of Example 2.

## 15 Example 4 (Composition example 3)

Four kinds of the compounds of the present invention (sample Nos. I, 2, 9 and I7) and the following two compounds which have a similar structure, but do not fall in the scope of the present invention:

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$$C_7H_{15} \longrightarrow O \longrightarrow OC_4H_9$$

(C  $\longrightarrow SC \longrightarrow N \longrightarrow I$ ) and

 $A = A_1S - C \longrightarrow A_2S - C$ 
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were blended together in equal quantities to prepare a liquid crystal composition.

This liquid crystal composition was smectic C composition and its phase transition points were as follows:

When this smectic C composition is blended with an optically active liquid crystal compound, it is possible to obtain superior chiral liquid crystal compositions as in the case of Example 2.

## 55 Claims

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1. Liquid crystal compounds of the formula:

$$R^{1} - O_{N} - O_{R}^{2}$$
 (I)

wherein R<sup>1</sup> represents a C<sub>7</sub>-C<sub>12</sub> alkyl group and R<sup>2</sup> represents a C<sub>5</sub>-C<sub>12</sub> alkyl group.

2. A smectic C liquid crystal composition consisting of a plurality of liquid crystal compound as claimed

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## in claim 1.

- 3. A smectic C liquid crystal composition comprising at least one liquid crystal compound as claimed in claim 1.
- 4. A chiral smectic (SC\*) liquid crystal composition comprising at least one liquid crystal compound as claimed in claim (I) and at least one kind of optically active liquid crystal compounds.
- 5. A chiral smectic C liquid crystal composition comprising a smectic C liquid crystal composition as claimed in claim 2 and at least one optically active liquid crystal compound.
- 6. A chiral smectic C liquid crystal composition comprising a smectic C liquid crystal composition as claimed in claim 3 and at least one optically active liquid crystal compound.
- 7. A light switching element utilizing ferroelectricity constituted by the use of a chiral smectic C liquid crystal composition comprising one of liquid crystal compound as claimed in claim 1 together with at least one optically active liquid crystal compound.



EPO Form 1503 03.82

## **EUROPEAN SEARCH REPORT**

EP 87 30 2634

Category	Citation of document wi	SIDERED TO BE RELEVA th indication, where appropriate,	Relevant	CLASSIFICATION OF THE
alogory	of rele	vant passages	to claim	APPLICATION (Int. Cl.4)
	formula III, li	(K.K. SUWA es 35-52; page 2, ne 62, page 6, 5 * & JP - A - 61	1,3	C 07 D 213/30 C 09 K 19/34 C 09 K 19/42 C 07 D 309/34
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# **EUROPEAN SEARCH REPORT**

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	DOCUMENTS CONS	Page 2		
Category	of rele	th indication, where appropriate, vant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
P,X	102, line 30;	(MERCK PATENT , lines 4-8; page page 103, lines page 104, lines	1-7	
P,X	formulas 2-5, pa	-9,10; page 23, ge 24, formulas line 7 - page 28,	1-7	
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